

# PATENT ABSTRACTS OF JAPAN

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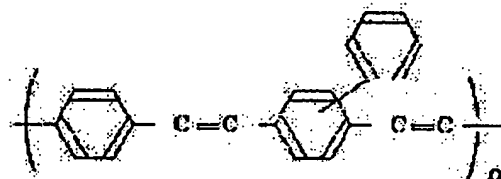
(72)Inventor : YAMAMOTO RYUICHI  
TAKAGI MASAKAZU

## (54) LINEAR POLY(PHENYLENE-ETHYNYLENE-BIPHENYLENE-ETHYNYLENE) POLYMER

(57)Abstract:

PURPOSE: To obtain a polymer having  $\pi$  conjugated bonds in the main chain, which can exhibit excellent conductivity and optical response as well as excellent heat resistance and chemical stability, is easily moldable because of its high solubility in an organic solvent, and is extensively used as a general-purpose functional material.

CONSTITUTION: A dihalobiphenyl and diethynylbenzene are subjected to a coupling reaction through dehydrohalogenation in the presence of a palladium- copper catalyst and an amine to obtain the objective polymer represented by the formula.



## LEGAL STATUS

[Date of request for examination] 02.05.2000

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[Patent number] 3198365

[Date of registration] 15.06.2001

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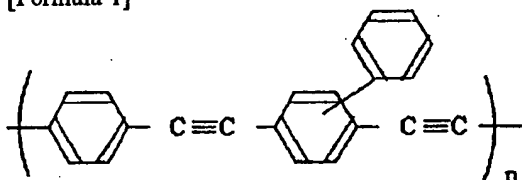
CLAIMS

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[Claim(s)]

[Claim 1] the line shown by the following formula -- the poly (phenylene-ethynylene-biphenylene-ethynylene) polymer

[Formula 1]

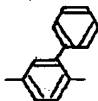


[Claim 2] the line according to claim 1 which consists of carrying out the dehydrohalogenation coupling reaction of the compound shown by the formula of the following-izing 2, and the compound (however, either Ar in both formulas or Ar' of biphenylene -2, 5-diyl, and another side being p-phenylene) shown by the formula of the following-izing 3 under a palladium-copper catalyst and amine presence -- the manufacture technique of the poly (phenylene-ethynylene-biphenylene-ethynylene) polymer

[Formula 2]



(式中、Xはハロゲンを表わし、Arは



または を表わす。)

[Formula 3]



(式中、Ar' は または を表わす。)

[Claim 3] a line according to claim 1 -- the luminescent material which consists of a poly (phenylene-ethynylene-biphenylene-ethynylene) polymer

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## DETAILED DESCRIPTION

## [Detailed Description of the Invention]

[0001]

[Field of the Invention] the line for which this invention is used as a heat-resistant functional material of light and electric responsibility -- it is related with the luminescent material using the poly (phenylene-ethynylene-biphenylene-ethynylene) polymer, its manufacture technique, and the aforementioned polymer

[0002]

[Description of the Prior Art] Generally a polyacetylene is made into the start as an example of the macromolecule which has pi conjugation combination in a principal chain. Although poly-arylene vinylenes, such as poly-arylenes, such as poly (p-phenylene), poly (2, 5-\*\*\*\*\*), or poly (1, 4-naphthalene diyl), poly (p-phenylene vinylene), or poly (2, 5-\*\*\*\*\* vinylene), etc. are mentioned Each of these makes the basic skeleton the repeat of -two-fold single bond combination. And a ring joins together continuously and it has the thermal resistance which whose above mentioned poly-arylene which has pi conjugation combination was chemically stable, and was excellent.

[0003] It is known that the conductivity about the same as a metal is shown by doping under a special condition about the polyacetylene of the easiest structure about such electric responsibility.

[0004] Moreover, pi conjugated-system macromolecule which has a triple bond in a principal chain has high crystallinity, and although the research as a nonlinear optics material is carried out about various poly-diacetylenes, there are very few research reports about other compounds. As a synthetic example of pi conjugated-system macromolecule which has an arylen-acetylenic linkage, poly (1, 4-phenylene-ethynylene) or poly (1, 4-phenylene-ethynylene -2, 5-\*\*\*\*\*-ethynylene) is in a principal chain.

[0005]

[Problem(s) to be Solved by the Invention] However, the above mentioned conventional conductive polyacetylene is lacking in fabricating-operation nature, and the trouble of being unstable is in it chemically. Moreover, the solubility over an organic solvent was low, and since almost all the compounds of the poly-arylene of the others to which conductivity is expected similarly were also non-\*\*, they were not fully able to aim at use as a functionality material in which each characteristic feature was employed efficiently, either. Since it went via a precursor \*\*\*\*\* to a solvent in the synthesis method and it was difficult to perform conversion from a precursor completely although especially the poly-arylene vinylene that has the interval-structure of a polyacetylene and a poly-arylene is considered to be useful as an expected material, it was what has the fault which a defect produces to a conjugate chain.

[0006] moreover, also in pi conjugated-system macromolecule which has an arylen-acetylenic linkage in a principal chain, as for these, that it is hard (Bull.Chem.Soc.Jpn., 57 volumes, 752 pages, (1984)) melts into an organic solvent -- it is -- it was difficult for the shape of the shape of a thin film, or yarn to carry out a fabricating operation to the configuration which forms and can employ functions, such as conductivity, efficiently

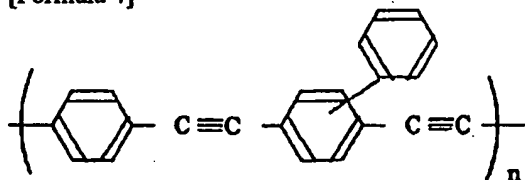
[0007] Then, it is making into the technical problem for this invention to have solved the above-mentioned trouble, to have shown conductivity and optical responsibility and to have raised the solubility to various kinds of organic solvents moreover while it held the thermal resistance and the chemical stability which were excellent in this thing by devising the structure of the molecule of a macromolecule of having pi conjugation combination in a principal chain, and it to be easy the fabricating operation and to fully have raised availability as a functionality material.

[0008]

[Means for Solving the Problem] the line shown by the formula of the following-izing 4 in this invention in order to solve the above-mentioned technical problem -- it considered as the poly (phenylene-ethynylene-biphenylene-ethynylene) polymer

[0009]

[Formula 4]



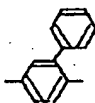
[0010] This polymer can be manufactured by carrying out the dehydrohalogenation coupling reaction of the compound shown by the formula of the following-izing 5, and the compound (however, either Ar in both formulas or Ar' of biphenylene -2, 5-diyl, and another side being p-phenylene) shown by the formula of the following-izing 6 under a palladium-copper catalyst and amine presence.

[0011]

[Formula 5]



(式中、Xはハロゲンを表わし、Arは



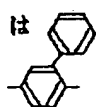
または を表わす。)

[0012]

[Formula 6]



(式中、Ar'は または を表わす。)



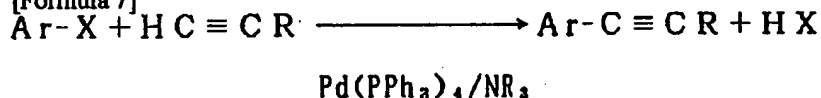
[0013] moreover, the above mentioned line -- it can also consider as the luminescent material which consists of a poly (phenylene-ethynylene-biphenylene-ethynylene) polymer Below, the detail is described.

[0014] the line shown in the above-ization 4 -- the principle of the manufacturing method of the poly (phenylene-ethynylene-biphenylene-ethynylene) polymer is explained below

[0015] First, if an aryl halide (Ar-X) and terminal acetylene are made to react under a palladium phosphine complex compound catalyst (Pd4 (PPh3)) and amine (NR3) presence as shown in the reaction formula of the following-izing 7, acetylene hydrogen will be replaced by the aryl group (for example, J.Organomet.Chem., 93 volumes, 253 pages, (1975)). in this case, aforementioned palladium-catalyst Pd (PPh3)4 -- replacing with -- PdCl2 -- 2 (PPh3), PdCl2 2 (DPPE) (DPPE shows \*\*\*\*\* phosphino ethane among a formula), and Pd(OAc)2+2PPh3 etc. -- the catalyst which consists of a palladium compound can also be used Moreover, as the aforementioned amine, it may be strong basic alkylamines, such as a diethylamine besides a triethylamine, and a piperidine, and especially the loadings do not have a limit and there should just be a little superfluously to a reaction substrate.

[0016]

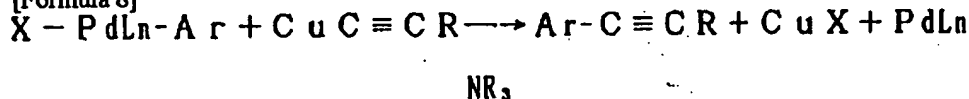
[Formula 7]



[0017] If copper iodide is added especially, a reaction will advance smoothly. It is because it is thought that a copper acetylide generates and a reaction advances by the formation [ palladium / (Pd) ] of transformer metal and reduction desorption as shown in the reaction formula of the following-izing 8. (TetrahedronLett., 4407 pages, 1975) . In this case, the aforementioned copper iodide of it replacing with this and the effect with the same said also of a copper chloride and a copper bromide especially being expected, although it is desirable is natural in respect of the ease of dealing with it.

[0018]

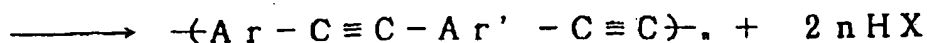
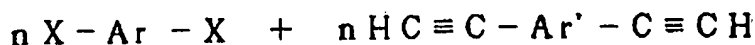
[Formula 8]



[0019] Therefore, if the dihalogen-ized aryl which has two halogens (X) in a molecule and which is an aromatic compound, and the diethynyl aryl which is the aromatic compound which has two ethynyl machines are made to react under a palladium-copper catalyst and amine presence with a solvent as shown in the reaction formula of the following-izing 9, a polymer will be obtained by dehydrohalogenation distributor shaft coupling.

[0020]

[Formula 9]



Pd/Cu/NR<sub>3</sub>

[0021] In addition, 0.1-10 mol% of the blending ratio of coal of the palladium catalyst used by the above-mentioned manufacturing method is desirable to a reaction substrate, and is desirable. [ especially 1-4 mol% of ] About [ 0.1-10mol% ] is suitable for the blending ratio of coal of copper iodide. Moreover, a reaction solvent makes toluene the example of representation, and it can be used for it, without limiting benzene, a dimethylformamide (it being hereafter written as DHF), a tetrahydrofuran (it being hereafter written as THF), especially a pyridine, etc. It may not limit especially about the concentration of the reaction substrate in a reaction solvent, either, and 0.01-1 mol /may be [ 1. ] 0.05-0.2 mols/l. especially preferably preferably. if reaction temperature may be the solvent reflux temperature (for example, 40-100 degrees C) from a room temperature, reaction time is 5 minutes - about 100 hours further again and it is about 1 - 24 hours especially -- a manufacture -- it is efficiently desirable

[0022]

[Example] It is tetrakis(triphenyl phosphine)palladium as 2, 5-dibromo biphenyl 0.5mmol, p-diethynyl benzene 0.5mmol, and a zero \*\* palladium compound :P d(PPh<sub>3</sub>) 4 2ml of triethylamines is added to 0.01mmol and copper iodide 0.015mmol, it stirred for 1 hour and 70 degrees C was made to react in 6ml of toluene. In the place where the powder-like polymerization object was obtained in this system of reaction, after a lot of methanols washed enough, it filtered and dried using the vacuum line. The yield of the obtained polymer was 77%.

[0023] When the component element by thermal decomposition was analyzed about the obtained polymerization object according to the conventional method, the percent which use carbon and hydrogen as a constituent element were 95.56% of carbon, and 4.44% of hydrogen.

[0024] The calculated value of elementary-composition C<sub>22</sub>H<sub>12</sub> of the polymer which, on the other hand, repeats the compound shown by the formula of the above-izing 4, and is made into a unit is 95.63% of carbon, and 4.37% of hydrogen, and was mostly in agreement with the percent of the aforementioned measured value. In addition, the polymerization object obtained the account of a front had the high thermal stability, and since it was not easy to carry out perfect combustion in case of elemental analysis, it was presumed that few errors have arisen in observed value and calculated value.

[0025] The infrared absorption spectrum of the [example 1 of experiment] example showed the following absorption (cm<sup>-1</sup>), respectively.

[0026] 3028, 2194, 1592, 1505, 1461, 1441, 1401, 1379, 1327, 1100, 1066, 1028, 1015, 894, 834, 762, 697,623,608,567, thus the infrared absorption spectrum of an example showed absorption characteristic of stretching vibration to the 2194cm<sup>-1</sup> neighborhood at the acetylenic linkage, and absorption of 834cm<sup>-1</sup> showed absorption characteristic of the deformation vibration outside C-H plane of an aromatic ring.

[0027] About the polymer of the [example 2 of experiment] example, when the thermogravimetric analysis was performed, pyrolysis temperature is about 300 degrees C, and showed the high thermal stability.

[0028] When the ultraviolet visible spectrum in a chloroform solution was measured about the [example 3 of experiment] example, the absorption maximum which shows comparatively acute and clear Yamagata to 368nm was shown.

[0029] the place which calculated number average molecular weight (Mn) about the polymer of the [example 4 of experiment] example according to the G.P.C method (tetrahydrofuran use) -- 3.37x10<sup>3</sup> it was . moreover, weight average molecular weight (Mw) -- 1.19x10<sup>4</sup> it was .

[0030] About the polymer of the [example 5 of experiment] example, it is 1mg/at about 25 degrees C. The solution of chloroform, N-methyl pyrrolidone (NMP), a tetrahydrofuran (THF), toluene, benzene, and dimethyl sulfoxide (DMSO) was prepared by the concentration of a milliliter, it extended on the laboratory dish, and the film-like matter was obtained by removing a solvent by the evaporating method after that.

[0031] When the infrared absorption spectrum of this matter was investigated, it was in agreement with the infrared absorption spectrum of a polymer before adding a solvent, and it became clear that the polymer of an example can fabricate easily [ the shape of yarn etc. ] by the shape of a film, the well-known radiation method, etc.

[0032] Moreover, when the polymer of an example was melted in 25-degree C chloroform (99% concentration) on stirring conditions, 11mg /ml ] solubility was shown.

[0033] [Example 6 of an experiment] Again, photogenesis was observed with as feeble as the semiconductor nature accompanied by applied voltage in what formed the thin film in the shape of a metal substrate by the vacuum deposition method about the polymer of an example, and what can be borne as a semiconductor material or a electron optics material at use was obtained.

[0034]

[Effect] the line shown by the predetermined formula as this invention was explained above, since it considered as the poly (phenylene-ethynylene-biphenylene-ethynylene) polymer While it becomes the compound by which continuous pi conjugated system to which the phenyl group and the biphenyl machine met the principal chain of a macromolecule is formed, and orientation was controlled highly and the outstanding thermal resistance and the outstanding chemical stability are held Moreover, it becomes

what shows conductivity and optical responsibility, since the solubility to an organic solvent is high, the fabricating operation is easy, and there is an advantage which can offer the very high thing of availability as a general-purpose functionality material.

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**Effect**

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**TECHNICAL PROBLEM**

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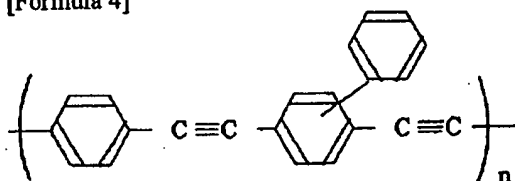
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## MEANS

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[0009]

[Formula 4]



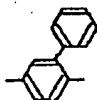
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[0011]

[Formula 5]



(式中、Xはハロゲンを表わし、Arは



または を表わす。)

[0012]

[Formula 6]



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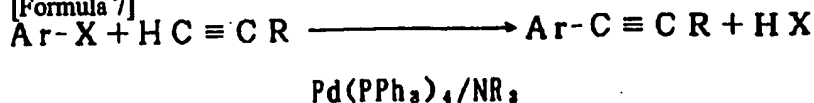
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[0015] First, if an aryl halide (Ar-X) and terminal acetylene are made to react under a palladium phosphine complex compound catalyst (Pd<sub>4</sub>(PPh<sub>3</sub>)<sub>4</sub>) and amine (NR<sub>3</sub>) presence as shown in the reaction formula of the following-izing 7, acetylene hydrogen will be replaced by the aryl group (for example, J. Organomet. Chem., 93 volumes, 253 pages, (1975)). in this case, aforementioned palladium-catalyst Pd(PPh<sub>3</sub>)<sub>4</sub> -- replacing with -- PdCl<sub>2</sub> · 2(PPh<sub>3</sub>), PdCl<sub>2</sub> 2(DPPE) (DPPE shows phosphino ethane among a formula), and Pd(OAc)<sub>2</sub> + 2PPh<sub>3</sub> etc. -- the catalyst which consists of a palladium compound can also be used Moreover, as the aforementioned amine, it may be strong basic alkylamines, such as a diethylamine besides a triethylamine, and a piperidine, and especially the loadings do not have a limit and there should just be a little superfluously to a reaction substrate.

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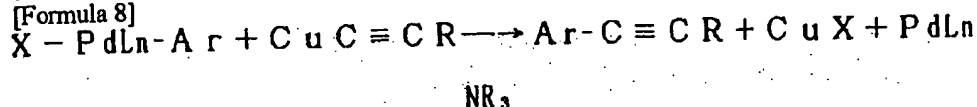
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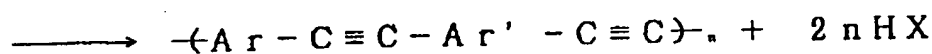
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[0020]

[Formula 9]



$\text{Pd/Cu/NR}_3$

[0021] In addition, 0.1-10 mol% of the blending ratio of coal of the palladium catalyst used by the above-mentioned manufacturing method is desirable to a reaction substrate, and is desirable. [ especially 1-4 mol% of ] About [ 0.1-10mol% ] is suitable for the blending ratio of coal of copper iodide. Moreover, a reaction solvent makes toluene the example of representation, and it can be used for it, without limiting benzene, a dimethylformamide (it being hereafter written as DHF), a tetrahydrofuran (it being hereafter written as THF), especially a pyridine, etc. It may not limit especially about the concentration of the reaction substrate in a reaction solvent, either, and 0.01-1 mol /may be [ 1. ] 0.05-0.2 mols/l. especially preferably preferably. if reaction temperature may be the solvent reflux temperature (for example, 40-100 degrees C) from a room temperature, reaction time is 5 minutes - about 100 hours further again and it is about 1 - 24 hours especially -- a manufacture -- it is efficiently desirable

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EXAMPLE

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[Example] It is tetrakis(triphenyl phosphine)palladium as 2, 5-dibromo biphenyl 0.5mmol, p-diethynyl benzene 0.5mmol, and a zero \*\* palladium compound :P d(PPh<sub>3</sub>) 4 2ml of triethylamines is added to 0.01mmol and copper iodide 0.015mmol, it stirred for 1 hour and 70 degrees C was made to react in 6ml of toluene. In the place where the powder-like polymerization object was obtained in this system of reaction, after a lot of methanols washed enough, it filtered and dried using the vacuum line. The yield of the obtained polymer was 77%.

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[0030] About the polymer of the [example 5 of experiment] example, it is 1mg/at about 25 degrees C. The solution of chloroform, N-methyl pyrrolidone (NMP), a tetrahydrofuran (THF), toluene, benzene, and dimethyl sulfoxide (DMSO) was prepared by the concentration of a milliliter, it extended on the laboratory dish, and the film-like matter was obtained by removing a solvent by the evaporating method after that.

[0031] When the infrared absorption spectrum of this matter was investigated, it was in agreement with the infrared absorption spectrum of a polymer before adding a solvent, and it became clear that the polymer of an example can fabricate easily [ the shape of yarn etc. ] by the shape of a film, the well-known radiation method, etc.

[0032] Moreover, when the polymer of an example was melted in 25-degree C chloroform (99% concentration) on stirring conditions, 11mg /ml ] solubility was shown.

[0033] [Example 6 of an experiment] Again, photogenesis was observed with as feeble as the semiconductor nature accompanied by applied voltage in what formed the thin film in the shape of a metal substrate by the vacuum deposition method about the polymer of an example, and what can be borne as a semiconductor material or a electron optics material at use was obtained.

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[Translation done.]